

B174

(19)



Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 0 708 798 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
26.04.2000 Bulletin 2000/17

(21) Application number: 94920792.2

(22) Date of filing: 21.06.1994

(51) Int. Cl.⁷: C08K 13/06, G02F 1/167,
G03G 17/04

(86) International application number:
PCT/US94/07042

(87) International publication number:
WO 95/02636 (26.01.1995 Gazette 1995/05)

(54) SUSPENSION FOR USE IN ELECTROPHORETIC IMAGE DISPLAY SYSTEMS

SUSPENSION ZUR VERWENDUNG IN ELEKTROPHORETISCHEN BILDANZEIGESYSTEMEN
SUSPENSION POUR SYSTEME DE VISUALISATION ELECTROPHORETIQUE

(84) Designated Contracting States:
BE DE FR GB IT NL

(30) Priority: 16.07.1993 US 92749

(43) Date of publication of application:
01.05.1996 Bulletin 1996/18

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(56) References cited:

US-A- 4 285 801

US-A- 4 298 448

US-A- 4 655 897

US-A- 4 889 603

US-A- 5 206 108

- JOURNAL OF APPLIED PHYSICS, Vol. 49, No. 9, published September 1978, MURAU et al.: "The Understanding and Elimination of Some Suspension Instabilities, in an Electrophoretic Display", pages 4820-4829.
- APPLIED SPECTROSCOPY, Vol. 33, No. 2, issued 1979, B. FITZHENRY: "Identification of a Charging Mechanism using Infrared Spectroscopy", pages 107-110, especially 108.

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Description

TECHNICAL FIELD OF THE INVENTION

5 [0001] The present invention relates to an electrophoretic display composition for display systems, electrostatic printing or the like and a corresponding method of manufacturing a suspension used therein. More particularly, the present invention relates to a suspension which utilizes steric stabilization to prevent pigment particle flocculation.

BACKGROUND ART

10 [0002] The electrophoretic effect is well known and the prior art is replete with a number of patents and articles which describe the effect. As will be recognized by a person skilled in the art, the electrophoretic effect operates on the principle that certain particles, when suspended in a medium, can be electrically charged and thereby caused to migrate through the medium to an electrode of opposite charge. Electrophoretic image displays (EPIDs) utilize the electrophoretic effect to produce desired images.

15 [0003] EPIDs generally comprise a suspension of colored charged pigment particles dispersed in a dyed solvent of contrasting color, which is injected into a cell consisting of two parallel and transparent conducting electrode panels. The charged particles are transported and packed against one electrode under the influence of an electric field, so that the viewer may see the color of the pigment. When the polarity of the field is reversed, the pigment particles are transported and packed on the opposite electrode. If the optical density of the dyed solvent is high enough to absorb the light scattered by the particles residing on the rear electrode, the observer will perceive the color of the dyed solvent. The performance of the resulting display is strongly dependent upon the suspension stability.

20 [0004] In non-aqueous dispersions colloid particles generally owe their stability to the fact that their surfaces are charged and, hence, repel each other. When the particles are uncharged, the dispersion is unstable. The fact that a colloidal particle bears a net surface charge is not a sufficient condition for stability because electroneutrality demands that the particle plus its immediate surroundings bear no net charge. In other words, the surface charge must be compensated by an equal but opposite counter charge, so that surface charge and countercharge together form an electrical double layer. P. Murau and B. Singer, in an article appearing in Vol. 49, No. 9 of the Journal of Applied Physics (1978) and entitled "The Understanding and Elimination of Some Suspension Instabilities in an Electrophoretic Display", indicate that when the double layer is compressed, the particles can approach each other to within a few hundred angstroms before repulsion is felt whereupon the van der Waals attraction becomes so strong that aggregation occurs.

25 [0005] The interactions of particle surfaces and charge control agents in colloidal suspensions has been the subject of considerable research. Reference is made to an article entitled "Mechanism of Electric Charging of Particles in Non-aqueous Liquids" appearing in Vol. 15 of the Journal of the American Chemical Society (1982), wherein F. M. Fowkes et al discuss the mechanism of electrostatic charging of suspended acidic particles by basic dispersants in solvents of low dielectric constant. Reference is also made to an article entitled "Steric and Electrostatic Contributions to the Colloidal Properties of Nonaqueous Dispersions" appearing in Vol. 21 of the Journal of the American Chemical Society (1984) wherein F.M. Fowkes and R. J. Pugh discuss the importance of anchoring sites for steric stabilizers in minimizing particle flocculation. The essential point developed by these references is that particle surface interactions are acid-base in character. Acidic pigment surface sites and basic charge control agents yield negative pigment surface charge. On the other hand, basic pigment surface sites and acidic charge control agents yield positive pigment surface charge.

30 [0006] Since electrophoretic devices utilize low polarity liquids in which ionization of ordinary organic adds and salts is negligible (approximately 10^{-10} moles per liter), the charge of the particle is governed by trace impurities unless otherwise controlled by adsorbing on the pigment surface a suitable charge control agent. This amount of charge, although sufficient for electrophoretic activity may still be inadequate for electrostatic stabilization of the suspension. If the charge control agent is also polymeric, or a polymeric dispersant is present in addition, the colloid stability can be further enhanced.

35 [0007] Over recent years, attention has therefore been directed to dispersion stabilization by way of adsorbed polymers on particle surfaces. If two colloidal particles coated with adsorbed layers of polymers approach each other, steric repulsion can occur as soon as the polymer layers start to penetrate. According to Murau and Singer, the polymer molecules adsorbed on a colloidal particle never lie flat on the surface. Rather, parts of the long chains (loose-ends, side branches, and loops) are free from the surface and surrounded by liquid. The overlapping of the polymer chains upon close approach can be pictured as a localized increase in the polymer concentration. This case is thermodynamically less favorable than the "dilute" situation existing when particles are far apart.

40 [0008] As will be recognized by a person skilled in the art, the selection of the electrophoretic particles used in the EPID is very important in determining the performance of the EPID and the quality of the viewed image produced. Ideally, electrophoretic particles should have an optimum charge/mass ratio, which is dependent upon the particle size and surface charge, in order to obtain good electrostatic deposition at high velocity as well as rapid reversal of particle

motion when voltages change. Additionally, it is desirable to utilize electrophoretic particles that have essentially the same density as the fluid medium in which they are suspended. By using electrophoretic particles of essentially the same density as the suspension medium, the migration of the electrophoretic particles through the medium remains independent of both the orientation of the EPID and the forces of gravity.

5 [0009] To effect the greatest optical contrast between electrophoretic particles and the suspension medium, it is desirable to have either light-colored particles suspended in a dark medium or black particles suspended in a back-lighted clear medium. In the prior art, it has been proven difficult to produce black electrophoretic particles that are dielectric, of uniform size and have a density matching that of a common suspension medium. As a result, EPIDs, commonly use readily manufactured light colored electrophoretic particles suspended in dark media. Such EPIDs are exemplified in U.S. Patent Nos: 4,655,897 to DiSanto et al., 4,093,534 to Carter et al., 4,298,448 to Muller et al., and 10 4,285,801 to Chaing. In such art, the light colored particles are commonly inorganic pigments. Titanium dioxide, for example, has been used in EPIDs to produce a good optical contrast between the white particles and the colored suspension medium. However, it has a density about 4 g/cm³ which is too high to match with any organic liquid to prevent the sedimentation problem. In the past decade, great effort has been made to solve the density problem of titanium dioxide. However, very little work has succeeded without trading off the quality of the images, especially in regard to the 15 whiteness. Coating titanium dioxide particles with a polymeric material to reduce the density of titanium dioxide is an example.

[0010] It is an object of the present invention to produce stable suspensions suitable for use in EPIDs, the suspension also having high electrophoretic sensitivity. It is a further object to produce light colored dielectric particles which 20 may be used in such suspensions.

DISCLOSURE OF THE INVENTION

[0011] The invention provides a working fluid for an electrophoretic image display device including a dispersion of 25 finely divided diarylide yellow pigment particles each having a charge of a given polarity and acidic hydrogen surface site in a suspension medium. Each of the pigment particles are transportable within the suspension medium, under the influence of an electric field, toward or away from at least one transparent plate. Adsorbed on the surface of each pigment particle is a thin film of a charge control agent having a polymer (e.g. polyisobutylene) chain and a basic anchoring 30 group which reacts with the acidic surface sites to form charge pairs therewith. The polymer chains extend from the pigment surfaces on which the charge control agent is adsorbed, thereby providing a steric barrier between pigment particles and preventing agglomeration. The specific gravity of the suspension medium is matched with that of the pigment particles by combining effective amounts of secondary butyl benzene and tetrachloroethylene. A solvent dye (e.g. blue) is added to absorb substantially all light scattered by the pigment particles when the pigments are transported away from the transparent plate being viewed.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the same becomes better understood by reference to the following detailed description when considered 40 in connection with the accompanying drawing wherein:

FIG. 1 is a diagrammatic, cross sectional view of an EPID having particles in accordance with the present invention; FIG.2 is a representation of the chemical structure of a stabilizer and charge control agent utilized in an EPID suspension prepared in accordance with the present invention; and 45 FIG. 3 is a representation of the chemical structure of a solvent dye which may be utilized in an EPID suspension prepared in accordance with the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

50 [0013] Although particles in accordance with the present invention can be used in many different applications where particles of yellow color and low density are desired, such as paint, ink and electrostatic toner, it is especially suitable for use in connection with electrophoretic image displays (EPID). Accordingly, the dielectric particles of the present invention will be described in connection with typical EPIDs.

[0014] Referring to Fig. 1, there is shown a cross-sectional view of a segment of a simple electrophoretic image display 10, magnified to show a single pixel (intersection). As will be recognized by a person skilled in the art, an EPID may 55 contain a volume of an electrophoretic dispersion 12 disposed between an anode 14 and a cathode 16. The anode 14 and cathode 16 are deposited upon glass plates 18, 20 in the form of a thin layer of indium-tin-oxide (ITO) or a like compound. The ITO layer is deposited in such a manner as to be substantially transparent when viewed through the glass

plates 18, 20.

[0015] With continuing reference to FIG. 1, the electrophoretic dispersion 12 comprises light colored electrophoretic particles 22 suspended in a dark colored medium 24. The electrophoretic particles 22 have a density substantially equivalent to that of the fluid medium 24 so as to remain randomly dispersed in the fluid medium 24, unaffected by the orientation of the EPID or the effects of gravity. When a sufficient electrical bias is applied between the anode 14 and cathode 16, the electrophoretic particles 22 migrate in response thereto to either the cathode 16 or anode 14 depending on polarity and displace the dark color medium 24 adjacent to the ITO layer, thereby creating a bright pixel. Reversing the voltage produces a dark pixel.

[0016] As noted previously, the production of light-colored image on a dark color background or a black image on a light background is highly desirable. However, a major obstacle to such a combination has been the lack of dielectric dark and light particles that have good hiding power and also a density that can be readily matched with common suspension fluids. In accordance with the present invention, the suspension is comprised of two yellow pigments which are density matched to, and dispersed in, a solvent medium of tetrachloroethylene and secondary butyl-benzene. The composition and physical properties of an exemplary suspension, in accordance with the present invention, for an electrophoretic display is depicted in Table I.

TABLE I

Diarylide Yellow (DY) pigment, #374-73, product of Sun Chemical Co.,	concentration	0.4% (by wt)
Diarylide Yellow (DY) pigment, #374-74B, product of Sun Chemical Co.	concentration	0.4%
Solvent Blue 35 (SB35) blue dye, #30,643-6, product of Aldrich Co., 1,4-Bis(butylamino)-9,10-anthracenedione	concentration	0.12%
OLOA 1200 stabilizer and charging agent, product of Chevron Chemicals Co.	concentration	0.4%
Tetrachloroethylene and secondary-butyl benzene, background solvent adjusted to a specific gravity of 1.43 g/ml	concentration	98.7%

[0017] As discussed earlier, pigment particles can be prevented from agglomerating if either an electrostatic or a steric baffler exists between particles. In accordance with the illustrative embodiment of the present invention, the particles 22 are selected from a diarylide yellow pigment group in which an acidic hydrogen site exists on the carbon atom bonded to two carbonyls and an azo group. Pigments Yellow 14 (Diarylide Yellow AAOT) and Yellow 55 (Diarylide Yellow AAPT) have this acid site and are used to prepare the novel suspension which is the subject of the present application.

[0018] As noted above, acidic pigment surface sites and basic charge control agents yield negative pigment surface charge. On the other hand, basic pigment surface sites and acidic charge control agents yield positive pigment surface charge. Since the diarylide yellows comprising the pigment particles utilize an acidic site, the charge control agents for these materials must be basic in character. Excellent results, for example, have been obtained using Chevron OLOA 1200 as the charge control and stabilizing agent. The chemical structure of Chevron OLOA 1200 is depicted in FIG. 2.

Although OLOA 1200 is the preferred charge control agent and stabilizer, other charge control agents, including basic barium sulfonate and several poly-vinyl pyridine block copolymers, may be utilized in effective amounts to achieve comparable results. OLOA 1200 is a polybutene succinimide with a basic anchoring group and a 5 nm (50 angstrom) long extended polyisobutylene chain. The long polyisobutylene chain allows OLOA to function as a steric stabilizer as well as a charge control agent. This dispersant is supplied as a 50 wt% solution in a mineral oil. It can be deoiled by adsorption from toluene onto silica with elution by acetone. The wt% of charge control agent given in Table I refers to the material as supplied. The basicity of OLOA 1200 has been evidenced by its interaction with the oil soluble acidic indicator due, Brom Phenol Magenta E (EK 6810) which is normally yellow but turns blue and then magenta with increasing basicity. The acidic form has an adsorption peak at 390 nm, the basic at 610 nm, and the isosbestic point is at 460nm. Attention has been focused on this molecules acid-base interaction in the aforementioned article by Murau and Singer and also in an article by B. Fitzhenry entitled "Identification of a Charging Mechanism using Infrared Spectroscopy" appearing in Applied Spectroscopy, Vol. 33, No. 2 (1979).

[0019] Besides reading with the pigment to form charge pairs, the OLOA 1200 molecule also forms a polymeric layer on the surface of the pigment particles which prevents particle flocculation. The state with the polymeric chains extending out from the particle's surface into the solvent is thermodynamically more stable than the state with the polymeric surface chains on adjoining particles intertwining with one another. This phenomenon is called steric stabilization.

[0020] Pigment motion in the suspension is not sufficient by itself to produce an image. A contrasting dye must be used to "hide" the pigment in dark areas of the image while the pigment itself produces the bright or yellow portion of the image. The pigment particles generate color by scattering light incident on them. While the particles scatter red, orange, yellow, and green light, the eye and brain of the observer integrate the various colors and the cumulative effect is a net yellow color. The dye, on the other hand, produces a color by absorbing all colors except blue or violet. This is a subtraction process. Accordingly, the ideal dye used in the display would have a sufficient optical density to absorb all of the light scattered by the pigment particles. In actual operation, some pigment scattered light will pass through the dye solution. However, the Solvent Blue 35 dye solution used in the illustrative embodiment of the present invention produces a very strong blue-black contrast to the yellow pigment. The chemical structure of the dye solution is depicted in FIG. 3. Similar anthraquinone dyes may also be used, for example, with other alkyl groups replacing the butyl group in Solvent Blue 35. An example is Oil Blue N, where the butyl group has been replaced by a pentyl group, can be used.

[0021] Another consideration in selecting the proper dye for the suspension is that the dye must not cause any detrimental side reactions therein. For example, interaction between dye and OLOA could produce highly mobile charged pairs, which are not visible and not properly regulated by the fields used to achieve image formation. The presence of such particles could severely and detrimentally affect fluid performance. Solvent Blue 35 has an amino group as its main reactive unit and can interact with the surface of the pigment without affecting fluid performance. Thus, selection of Solvent Blue 35 avoids any risk of reaction with the OLOA 1200, which has the same functional group.

[0022] The solvents utilized in the suspension of the present invention are tetrachloroethylene and sec-butyl benzene. Both compounds have high resistivity, a low dielectric constant, and are stable in electrical fields in the megavolt/meter range. The high specific gravity of tetrachloroethylene, 1.62 g/ml, allows specific gravity balancing to 1.43 g/ml with the less dense sec-butyl benzene. Further, neither solvent attacks the pigment at operating conditions. Another reason for the solvent choice is to arrive at a suspension viscosity which allows smooth pigment motion in the range of the applied field employed. At 20°C, the 1.43 g/ml specific gravity adjusted fluid has a viscosity of 1.4 mPa · s (1.4 centipoise). This proves to be a good central value to yield coordinated pigment motion over a range of temperatures from below 0°C to 40°C.

[0023] Besides materials which are intentionally measured out and included in the suspension of the present invention, there are other materials which must also be considered. Trace impurities of polar materials, especially water, detrimentally effect the stability of suspensions in non-polar solvents. For example, water adsorbs on the particle surface and tends to affect not only the magnitude but also the sign of the particle mobility. Mobility measurements indicate that efforts to remove adsorbed surface water from Pigment DY-14 #374-73, by heating it in a vacuum oven prior to suspension preparation, lead to a decrease in pigment particle mobility in the final suspension. This is in comparison to Pigment 14 #374-73 which was allowed to equilibrate in a 51% humidity chamber prior to suspension preparation. A similar effect is not noted for Pigment DY 14 #374-74B.

[0024] As seen in Table 1, the two yellow pigments utilized in the suspension of the present invention are the same Yellow 14 diarylide molecule. Pigment #374-74B has been treated with two surface active agents, hexadecane and aerosol C61. The surface active agents were present in the reaction mixture of the coupling reaction which formed the pigment molecule. The pigment #374-74B, when used as the exclusive pigment particle, produces suspensions with uncoordinated, turbulent particle motion. Suspensions using pigment #374-73 exclusively, on the other hand, produces suspensions with coordinated, smooth but slower, motion. Pigment #374-73 suspensions are also slower to move from an electrode when voltages are switched. Mixing these two pigments in a 50:50 ratio produces suspensions far superior for EPIDs than those made from either pigment alone.

[0025] The following example illustrates the process of making an EPID suspension in accordance with the present invention.

EXAMPLE I

[0026] The suspension is prepared by ballmilling all components for several hours in a teflon jar. This is done to break up the dry agglomerated pigment powder into individual particles and allow the exposed surface to interact with the stabilizer. The inner chamber of the teflon jar is essentially cylindrical in shape. Controlled humidity and temperature are preferred but not necessary. During milling the temperature of the suspension rises to a little over 40°C. The media used are 2.0 mm zirconium beads having a density of 6.0 g/ml. Prior to placement in the jars, the beads are milled twice for 30 minutes in a tetrachlorethylene solvent. After each milling, the solvent is strained off and discarded. Three different size jars and bead charges have been tested in which the twice-milled beads plus the suspension fill the jars approximately half-way. The specific test conditions are set forth in Table II.

TABLE II

JAR HEIGHT	JAR DIAMETER	BEAD CHARGE	SUSPENSION SAMPLE SITE
48mm	61mm	200g	50g
68mm	81mm	400g	100g
113mm	81mm	800g	200g

[0027] The mill employed is a commercially available paint shaker Model 5410 manufactured by the Red Devil Company. Adapters were placed in the shaker cells to hold the jars. The motion of the mill is in two dimensions. It rotates about 20° in the YZ plane while it moves side to side approximately 15mm in the X direction. The motion frequency is about 5 cycles per second. The duration of the entire milling process is approximately eight hours.

[0028] During the milling procedure, some of the OLOA-1200 is adsorbed into the surface of the beads, forming a coating thereon. It has been found that recycled beads tend to produce higher current mixes than new beads. After milling, the suspension is strained from the beads. At this point, the specific gravity of the mix is measured. If necessary, it is adjusted to make sure it is slightly more dense than the pigment. The suspension is divided into centrifuge tubes and spun at 5000 RPM for 30 minutes. the suspension is transferred to fresh test tubes by carefully drawing out the pigment and most of the liquid. A small amount of liquid and grey-weight dense solid, which has settled out, are left behind. The primary source of the residue is the zirconium oxide. The process of transferring to fresh tubes is repeated three times to ensure adequate removal of unwanted particulate matter.

[0029] The specific gravity of the suspension is now adjusted to 1.43 g/ml. Approximately 15ml of suspension, is exposed to an ultrasonic probe for one minute. The probe employed is a Model UP150 manufactured by Sonacor, which probe operates at 40 kHz and has a maximum power of 1,000 watts. The probe is operated at half-maximum power, which is sufficient to encourage adequate bubble formation, thereby driving out dissolved gasses. The liquid is warmed slightly by this process as well. Filling of the cell should occur as soon as possible after the ultrasonic treatment. Before use in an EPID cell, a portion of the suspension was placed in a diode and the pigment observed moving back and forth therein. At approximately 1.5 Hz, the mixture maintains uniform texture. With the pigment on one electrode of the diode in a field of around 600,000 volts/meter, the steady state current after 30 seconds is measured. A current density of about 4.4×10^{-7} amps/cm² was recorded.

[0030] A specially configured EPID test cell was constructed with means for applying timed voltage pulses thereacross. A 10 x 16 x 0.035 cm (4.5" x 6.4" x 0.014") cell is filled with 15 ml of the suspension and pulses are applied to establish the minimum length of pulse required to move the mix far enough away from one electrode so that the complete transition from yellow to black is observed. In a 5 by 7.5 cm (2" by 3") diode at approximately 600,000 volts/meter, the pulse length is 80 msec.

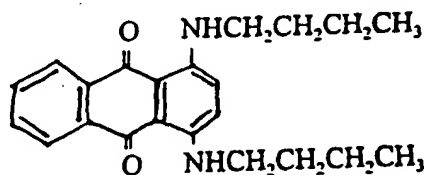
[0031] The electrophoretic composition in accordance with the present invention provided good overall visual performance and had a very fast response time, particularly during release of the pigment particles from the viewing electrode.

Claims

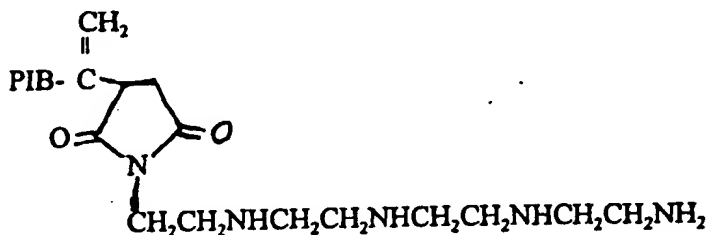
1. An electrophoretic display composition comprising:

a suspension medium comprising tetrachloroethylene and sec-butyl benzene;
 charged pigment particles suspended in said suspension medium, 50 wt. % of said pigment particles having
 been formed by a coupling reaction in the presence of at least one surface active agent, said suspension
 medium having a specific gravity corresponding to that of said pigment particles, said pigment particles defin-
 ing surfaces having at least one acidic hydrogen site per molecule and said pigment particles being selected
 from the group consisting of Pigment Yellow 14 and Pigment Yellow 55;
 a solvent dye dissolved in said suspension medium; and
 a charge control agent, said charge control agent being adsorbed on said pigment particles and having a basic
 anchoring group for reacting with said pigment particles to form charge pairs therewith and having a polymer
 chain extending therefrom, thereby providing a steric barrier between particles.

2. A composition according to claim 1, wherein the specific gravity of said suspension medium is 1.43 g/ml.
3. A composition according to claim 2, wherein said suspension medium is present in a concentration of 98.7% by weight.
4. A composition according to claim 1, wherein said solvent dye has the structure:



5. A composition according to claim 4, wherein said solvent dye is present in a concentration of 0.12% by weight.
6. A composition according to claim 1, wherein the pigment particles are Pigment Yellow 14.
7. A composition according to claim 6, wherein said pigment particles are present in said composition in a concentration of 0.8% by weight.
8. A composition according to claim 1, wherein said charge control agent is an amine having the structure:



where PIB is a polyisobutylene chain having an effective length of at least 5 nm (50 Å).

9. A composition according to claim 8, wherein said charge control agent is present in a concentration of 0.4% by weight.
10. A process for forming an electrophoretic display composition comprising the steps of:

forming a first plurality of discrete organic pigment particles by a coupling reaction in the presence of at least one surface active agent, each of said first plurality of pigment particles defining a surface ;

providing a second plurality of said discrete organic pigment particles which has not been treated with a surfactant, each of said second plurality of pigment particles defining a surface;

dispersing said first and second plurality of discrete organic pigment particles in a weight ratio of 50:50 in a suspension medium of tetrachloroethylene, each surface having at least one acidic hydrogen site and said first and second plurality of particles being selected from the group consisting of Pigment Yellow 14 and Pigment Yellow 55;

providing a charge control agent having at least one basic anchoring group in said suspension medium; adsorbing said charge control agent on the surfaces of said first and second plurality of particles and reacting said acidic hydrogen sites with said basic anchoring groups, thereby forming charge pairs and polymer chains extending from each surface to provide a steric barrier between the particles of both said first and second plurality of particles;

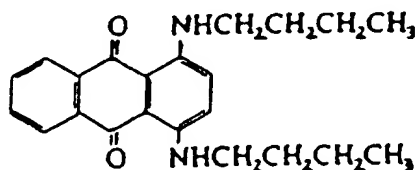
dissolving a solvent dye in said suspension medium; and

adding an effective amount of secondary-butyl benzene to said medium to obtain a specific gravity of said suspension medium which is the same as that of said first and second plurality of pigment particles.

11. The process according to claim 10, wherein said adsorbing step comprises ball milling said first and second plurality of pigment particles and said charge control agent together for at least two hours.

12. The process according to claim 10, wherein said dissolving step is performed prior to said adsorbing step and wherein said adsorbing step comprises ballmilling said first and second plurality of pigment particles and said charge control agent together for at least eight hours.

13. The process according to claim 10, wherein said solvent dye has the structure:



14. The process according to claim 10, further including the step of exposing said suspension medium to an ultrasonic probe to drive out any dissolved gasses.

Patentansprüche

1. Zusammensetzung für elektrophoretisches Display bzw. Bildanzeige, welche umfaßt:

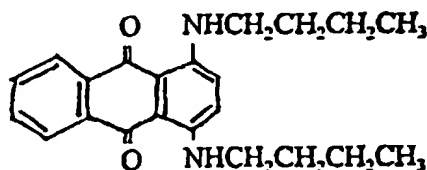
ein Tetrachloräthylen und sekundäres Butylbenzol enthaltendes Suspensionsmedium;

in diesem Suspensionsmedium suspendierte geladene Pigmentteilchen, wobei 50 Gew.% dieser Pigmentteilchen durch eine Kupplungsreaktion in Gegenwart wenigstens eines oberflächenaktiven Stoffs gebildet sind, das Suspensionsmedium ein dem spezifischen Gewicht der Pigmentteilchen entsprechendes spezifisches Gewicht besitzt, die Pigmentteilchen Oberflächen mit wenigstens einer sauren Wasserstoff-Stelle je Molekül aufweisen und die Pigmentteilchen aus der aus Pigment Yellow 14 und Pigment Yellow 55 bestehenden Gruppe gewählt sind;

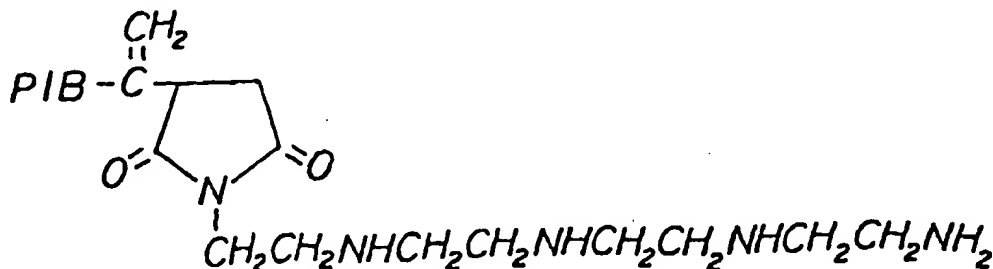
einen in dem Suspensionsmedium gelösten Lösungsmittelfarbstoff; sowie

ein Ladungssteuer- bzw.-kontrollmittel, das an den Pigmentteilchen adsorbiert ist und eine basische Ankergruppe für eine Reaktion mit den Pigmentteilchen unter Bildung von Ladungspaaren mit diesen aufweist und eine sich von dem Ladungssteuermittel aus erstreckende Polymerkette besitzt und dadurch eine sterische Barriere zwischen den Teilchen bildet.

2. Zusammensetzung nach Anspruch 1, in welcher das spezifische Gewicht des Suspensionsmediums 1.43 g/ml beträgt.
3. Zusammensetzung nach Anspruch 2, in welcher das Suspensionsmedium in einer Konzentration von 98.7 Gew.% vorliegt.
4. Zusammensetzung nach Anspruch 1, in welcher der genannte Lösungsmittelfarbstoff die folgende Struktur besitzt:



5. Zusammensetzung nach Anspruch 4, in welcher der genannte Lösungsmittel-Farbstoff in einer Konzentration von 0.12 Gew.% vorliegt.
6. Zusammensetzung nach Anspruch 1, in welcher die Pigmentteilchen das Pigment Yellow 14 sind.
7. Zusammensetzung nach Anspruch 6, in welcher die Pigmentteilchen in einer Konzentration von 0.8 Gew.% vorliegen.
8. Zusammensetzung nach Anspruch 1, in welcher das genannte Ladungssteuer- bzw. -kontrollmittel ein Amin mit der Struktur:



ist, wobei PIB eine Polyisobutylen-Kette mit einer effektiven Länge von wenigstens 5 nm (50 Angström) ist.

9. Zusammensetzung nach Anspruch 8, in welcher das Ladungssteuer- bzw. -kontrollmittel in einer Konzentration von 0.4 Gew.% vorliegt.
10. Verfahren zur Herstellung einer Zusammensetzung für ein elektrophoretisches Display bzw. Bildanzeige, wobei das Verfahren die folgenden Schritte umfaßt:

Erzeugen einer ersten Vielzahl diskreter organischer Pigmentteilchen durch eine Kupplungsreaktion in Gegenwart wenigstens eines oberflächenaktiven Stoffs, wobei jedes Pigmentteilchen dieser ersten Vielzahl von Pigmentteilchen jeweils eine definierte Oberfläche aufweist;

Bildung einer zweiten, nicht mit einem oberflächenaktiven Mittel behandelten Vielzahl diskreter organischer Pigmentteilchen, wobei jedes Pigmentteilchen dieser zweiten Vielzahl von Pigmentteilchen jeweils eine definierte Oberfläche aufweist;

Dispergieren der ersten und der zweiten Vielzahl diskreter Pigmentteilchen in einem Gewichtsverhältnis von

50 : 50 in einem Suspensionsmedium aus Tetrachloräthylen, wobei jede Oberfläche jeweils wenigstens eine saure Wasserstoff-Stelle aufweist und die erste und zweite Vielzahl von Teilchen aus der aus Pigment Yellow 14 und Pigment Yellow 55 bestehenden Gruppe gewählt werden;

5 Bilden eines Ladungssteuer- bzw. -kontrollmittels mit wenigstens einer basischen Ankergruppe in dem Suspensionsmedium;

10 Adsorbieren des Ladungssteuer- bzw.-kontrollmittels an den Oberflächen der ersten und zweiten Teilchenvielfzahl und Reaktion der genannten sauren Wasserstoff-Stellen mit den genannten basischen Ankergruppen, unter Bildung von Ladungspaaren und sich von jeder der Oberflächen aus erstreckender Polymerketten, zur Schaffung einer sterischen Barriere zwischen den Teilchen sowohl der ersten und der zweiten Teilchenvielfzahl;

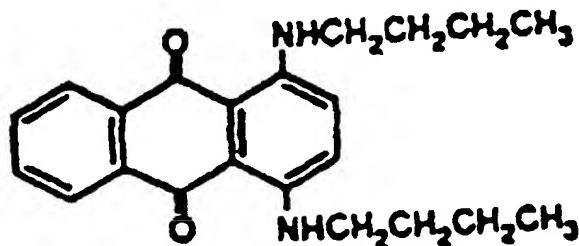
15 Auflösen eines Lösungsmittel-Farbstoffs in dem Suspensionsmedium; sowie

Zugabe einer wirksamen Menge von sekundärem Butylbenzol zu dem Medium, zur Erzielung eines spezifischen Gewichts des Suspensionsmediums, das mit dem spezifischen Gewicht der ersten und der zweiten Vielzahl von Pigmentteilchen im wesentlichen übereinstimmt.

20 11. Verfahren nach Anspruch 10, bei welchem der Adsorptionsschritt das gemeinsame Mahlen der ersten und der zweiten Pigmentteilchenvielfzahl und des Ladungssteuer- bzw. -kontrollmittels in der Kugelmühle während wenigstens zwei Stunden umfaßt.

25 12. Verfahren nach Anspruch 10, bei welchem der Verfahrensschritt des AuflöSENS vor dem Adsorptionsschritt ausgeführt wird und bei welchem der Adsorptionsschritt das gemeinsame Mahlen der ersten und zweiten Pigmentteilchenvielfzahl und des Ladungssteuer-bzw.-kontrollmittels in der Kugelmühle während wenigstens acht Stunden umfaßt.

30 13. Verfahren nach Anspruch 10, bei welchem der Lösungsmittel-Farbstoff die folgende Struktur besitzt:



45 14. Verfahren nach Anspruch 10, welches des weiteren den Verfahrensschritt umfaßt, in welchem das Suspensionsmedium einer Ultraschall-Sonde zum Austreiben jeglicher gelösten Gase ausgesetzt wird.

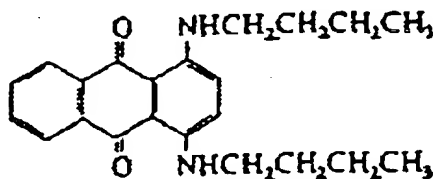
Revendications

50 1. Composition électrophorétique d'affichage comprenant :

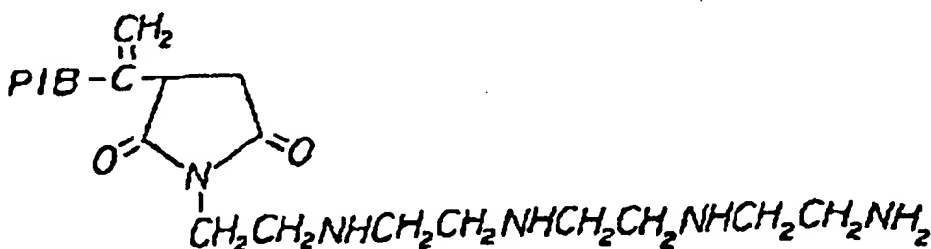
55 un milieu de suspension comprenant du tétrachloroéthylène et du sec-butyl benzène ;
des particules de pigment chargées suspendues dans le milieu de suspension, 50% en poids des particules de pigment ayant été formées par une réaction de couplage en présence d'au moins un agent tensioactif, le milieu de suspension ayant une gravité spécifique qui correspond à celle des particules de pigment, les particules de pigment définissant des surfaces ayant au moins un site d'hydrogène acide par molécule et les particules de pigment étant choisies dans le groupe comprenant le pigment Jaune 14 et le pigment Jaune 55 ;
un colorant solvant dissous dans le milieu de suspension ; et
un agent de contrôle de charge, cet agent de contrôle de charge étant adsorbé sur les particules de pigment,

ayant un groupe d'ancrage basique pour réagir avec les particules de pigment pour former des paires de charge avec celles-ci et une chaîne polymère s'étendant à partir de celles-ci, fournissant ainsi une barrière stérique entre particules.

2. Composition selon la revendication 1, dans laquelle la gravité spécifique du milieu de suspension est de 1,43 g/ml.
3. Composition selon la revendication 2, dans laquelle le milieu de suspension est présent selon une concentration de 98,7 % en poids.
4. Composition selon la revendication 1, dans laquelle le colorant solvant a la structure :



5. Composition selon la revendication 4, dans laquelle le colorant solvant est présent dans une concentration de 0,12% en poids.
6. Composition selon la revendication 1, dans laquelle les particules de pigment sont du pigment Jaune 14.
7. Composition selon la revendication 6, dans laquelle les particules de pigment sont présentes dans la composition selon une concentration de 0,8% en poids.
8. Composition selon la revendication 1, dans laquelle l'agent de contrôle de charge est une amine ayant la structure suivante :



où PIB est une chaîne de polyisobutylène ayant une longueur effective d'au moins 5 nm (50 Å).

9. Composition selon la revendication 8, dans laquelle l'agent de contrôle de charge est présent à une concentration de 0,4% en poids.
10. Procédé de formation d'une composition d'affichage électrophorétique comprenant les étapes suivantes :

former une première pluralité de particules discrètes de pigment organique par une réaction de couplage en présence d'au moins un agent tensio-actif, chacune de la première pluralité de particules de pigment définissant une surface ;
prévoir une seconde pluralité de particules discrètes de pigment organique qui ne sont pas traitées par un surfactant, chacune de la seconde pluralité de particules de pigment définissant une surface ;

dispenser les première et seconde pluralités de particules discrètes de pigment organique selon un rapport pondéral de 50/50 dans un milieu de suspension de tétrachloroéthylène, chaque surface comportant au moins un site d'hydrogène acide et les première et seconde pluralités de particules étant choisies dans le groupe comprenant le pigment Jaune 14 et le pigment Jaune 55 ;

prévoir un agent de contrôle de charge ayant au moins un groupe d'ancrage basique dans le milieu de suspension ;

adsorber l'agent de contrôle de charge sur les surfaces des première et seconde pluralités de particules et faire réagir les sites d'hydrogène acide avec les groupes d'ancrage basiques, formant ainsi des paires de charge et des chaînes de polymères s'étendant à partir de chaque surface pour former une barrière stérique entre les particules des première et seconde pluralités de particules ;

dissoudre un solvant dans le milieu de suspension ; et

ajouter une quantité active de butyl benzène secondaire au milieu pour obtenir une gravité spécifique du milieu de suspension qui est sensiblement identique à celle des première et seconde pluralités de particules de pigment.

11. Procédé selon la revendication 10, dans lequel l'étape d'adsorption comprend le broyage au boulet des première et seconde pluralités de particules de pigment et de l'agent de contrôle de charge, ensemble, pendant au moins deux heures.

12. Procédé selon la revendication 10, dans lequel l'étape de dissolution est réalisée avant l'étape d'adsorption et dans lequel l'étape d'adsorption comprend le broyage au boulet des première et seconde pluralités de particules de pigment et de l'agent de contrôle de charge, ensemble, pendant au moins huit heures.

13. Procédé selon la revendication 10, dans lequel le colorant solvant a la structure :



14. Procédé selon la revendication 10, comprenant en outre l'étape consistant à exposer le milieu de suspension à une sonde à ultrasons pour éliminer tout gaz dissous.

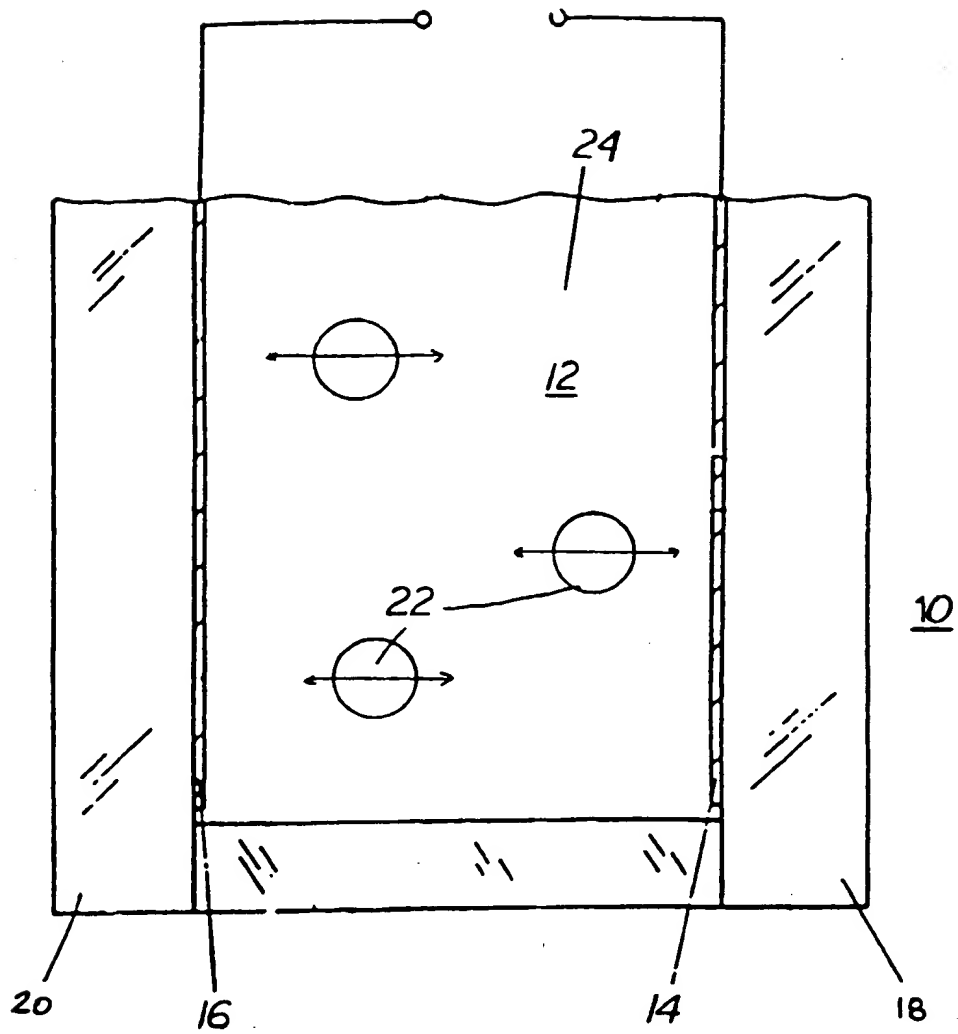


FIG.1

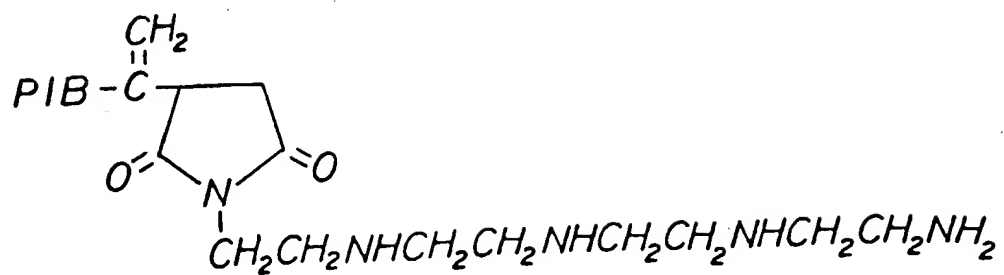


FIG. 2

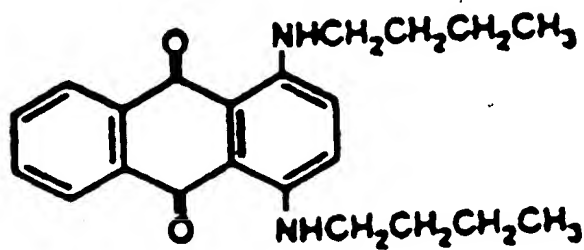


FIG. 3